

**SNL/SRNL Joint Project on degradation of mechanical properties  
in structural metals and welds for GTS reservoirs**

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***Project Background and Scope***

Stainless steel gas transfer system reservoirs are used to store hydrogen gas isotopes at high pressure; future gas transfer system designs will include aluminum alloy structural reservoirs and potentially additively-manufactured components. During service hydrogen isotopes dissolve into metals and reduce their fracture resistance. The degradation of the properties, however, is not uniform as the effects of hydrogen isotopes are highly dependent on the local microstructure as well as the applied and residual stresses (the latter due to manufacturing and welding). Additionally, isotope-induced fracture has a strong aging component: properties degrade continuously over the life of the product. The weld fusion zones and weld heat-affected zones are believed to be the most sensitive regions to isotope-induced fracture, although data for the as-manufactured base metal as well as weld microstructures is limited. Moreover, the effects of temperature over the entire STS range have not been comprehensively evaluated (low-temperature tends to enhance the negative effects of hydrogen) and superimposed residual stresses in finished components are generally unknown and reservoir specific.

This project was intended to enable SNL-CA to produce appropriate specimens of relevant stainless steels for testing and perform baseline testing of weld heat-affected zone and weld fusion zone. One of the key deliverables in this project was to establish a procedure for fracture testing stainless steel weld fusion zone and heat affected zones that were pre-charged with hydrogen. Following the establishment of the procedure, a round robin was planned between SNL-CA and SRNL to ensure testing consistency between laboratories. SNL-CA and SRNL would then develop a comprehensive test plan, which would include tritium exposures of several years at SRNL on samples delivered by SNL-CA. Testing would follow the procedures developed at SNL-CA. SRNL will also purchase tritium charging vessels to perform the tritium exposures. Although comprehensive understanding of isotope-induced fracture in GTS reservoir materials is a several year effort, the FY15 work would enabled us to jump-start the tests and initiate long-term tritium exposures to aid comprehensive future investigations. Development of a procedure and laboratory testing consistency between SNL-CA and SNRL ensures reliability in results as future evaluations are performed on aluminum alloys and potentially additively-manufactured components.

## Project Accomplishments

### *Development of Test Procedure*

A procedure was developed at SNL-CA to test hydrogen precharged bend specimens of two GTS-relevant alloys (21Cr-6Ni-9Mn and 304L forged stainless steels). Testing was focused on areas believed to be the most susceptible to hydrogen damage, e.g. weld fusion zone and heat affected zones. The procedure developed was tailored to improve the consistency and reliability of the fracture thresholds measured of hydrogen-isotope precharged welds and heat affected zones. Establishment of a procedure at SNL-CA in hydrogen-precharged samples was necessary in order to expedite and ensure quality testing of tritium-exposed samples at SRNL. A brief summary of the development of the test procedure is outlined below as it pertains to the overall purpose of generating reliable fracture data.

Bend testing was selected for this project as rectangular 3-point bend specimens could be extracted from forged stainless steel rings that were joined using gas tungsten arc (GTA) welding as shown in Fig. 1. The resulting microstructures of the weld fusion zone (FZ) and heat affected zone (HAZ) are shown in Figs. 2a and 2b, respectively. Fracture threshold tests on the weld FZ were performed by positioning a machined notch in the center of the weld, propagating a precrack, and performing the fracture test. Evaluating the fracture threshold of the weld HAZ presented a challenge as the region of the HAZ that exhibits grain growth is fairly limited as shown in Fig. 2b. Modifications to the weld geometry were needed to consistently terminate the precrack in the HAZ.

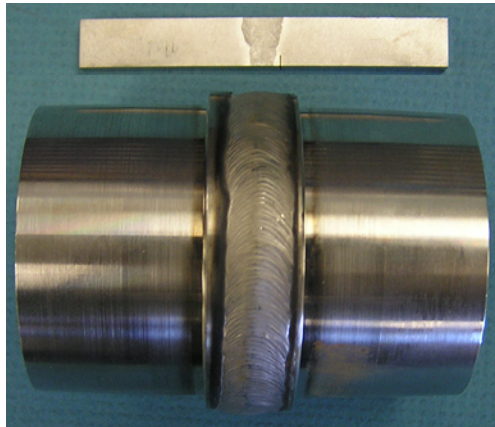


Figure 1 – Image of 21-6-9/308L weld ring and rectangular 3-point bend specimen removed from weld ring. Bend specimen was etched to reveal the weld.

A modified weld joint geometry was developed in collaboration with the GTS welding lab in SNL-CA to create a finished weld joint in which the weld fusion zone boundary line was parallel to the crack growth direction. The intent of this modification was to improve the repeatability of terminating a precrack in the HAZ of the weld. A weld fabricated using the modified weld joint can be observed in Fig. 3a as compared to the conventional weld joint shown in Fig. 3b. In both samples, a notch is located offset to the weld such that a precrack extended from the notch would propagate into the HAZ. The conventional weld joint consists of two J-edge weld pieces, which results in a tapered interface on both sides as shown in Fig. 3b. The modified weld joint consists of a J-edge and square edge. The resulting weld shown in Fig. 3a exhibits a taper on the left side but the square joint on the right side of the weld results in a weld boundary line that is close to parallel to the notch. Extending a precrack from the notch in Fig. 3a has a higher

probability of terminating in the HAZ than in Fig. 3b. Use of the modified weld joint as shown in Fig. 3a proved to be invaluable as the precrack was consistently observed to terminate in the HAZ as shown in Fig. 3c, indicated by the white arrow. This is important as fracture tests could then be conducted on the HAZ with assurance that the crack would remain in the HAZ region.

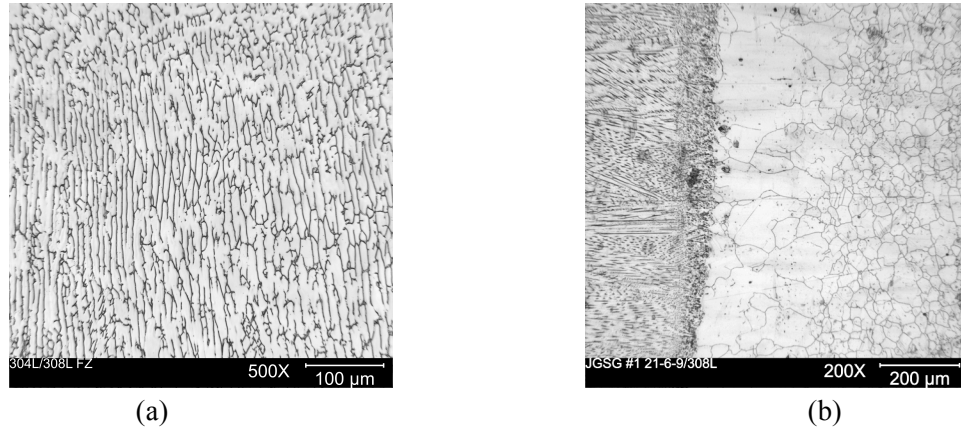


Figure 2 – (a) Optical micrograph of weld fusion zone of 304L/308L weld. (b) Heat affected zone of 21-6-9/308L showing enlarged grains as compared to base metal on the right.

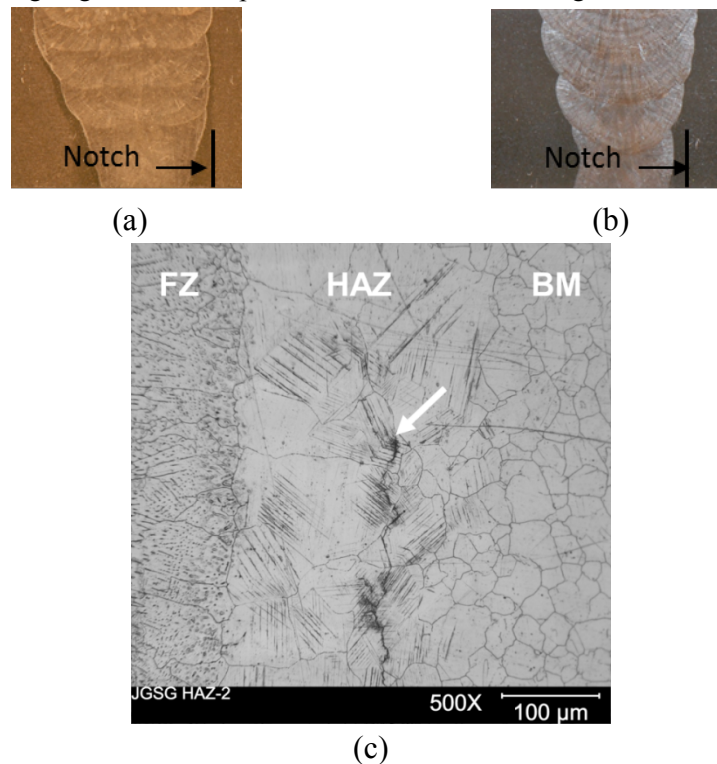


Figure 3 Micrograph of GTA welds fabricated from (a) modified weld joint, (b) conventional weld joint, (c) modified weld joint with precrack terminated in the HAZ. The white arrow points to the precrack termination point which is located in the HAZ.

The effects of testing rate on subcritical cracking thresholds were evaluated for the stainless steel weld FZ and HAZ and are shown in Fig. 4. The fracture thresholds were observed to decrease as the loading rate

decreased. At testing rates ( $dK/dt$ ) below  $0.1 \text{ MPa m}^{1/2} \text{ s}^{-1}$ , the decrease in fracture thresholds appeared to diminish for each material region tested and replicate tests showed repeatability. Therefore, the testing rate of  $0.1 \text{ MPa m}^{1/2} \text{ s}^{-1}$  was chosen as the critical testing rate which correlates to a displacement rate of approximately  $0.02 \text{ mm/min}$ . Performing tests at  $0.02 \text{ mm/min}$  strikes a balance between measuring lower bound fracture thresholds and improving testing efficiency. Testing at lower rates contribute to protracted testing times, whereas testing at higher rates can result in greater variability and non-conservative fracture thresholds. Post-test analysis of several HAZ tests confirmed that the crack remained in the HAZ for the duration of the fracture test as shown in Fig. 5. A more detailed summary of the test procedures can be found in [1].

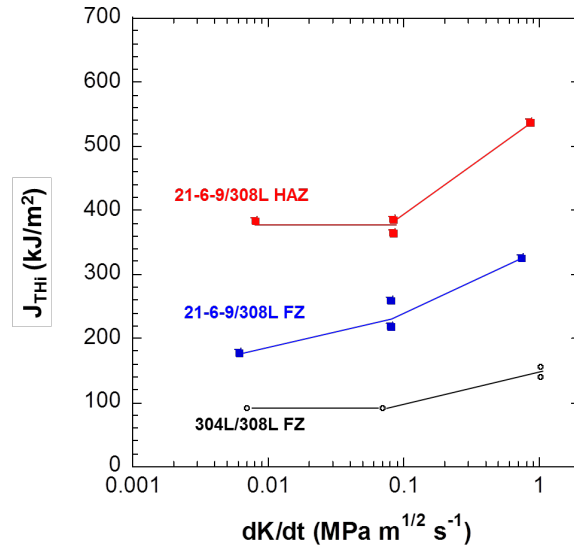


Figure 4 – Subcritical cracking threshold ( $J_{THI}$ ) as a function of testing rate (e.g. stress intensity factor rate,  $dK/dt$ ) for 21-6-9/308L HAZ, 21-6-9/308L FZ, and 304L/308L FZ.

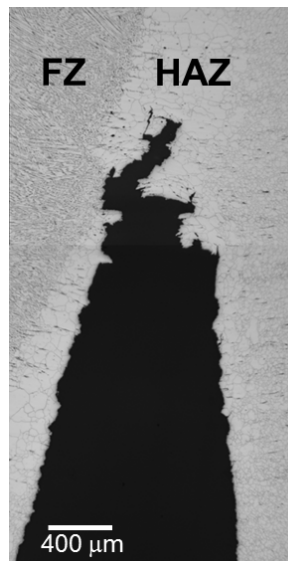


Figure 5 – Micrograph of crack profile after the subcritical cracking fracture test which demonstrates that the crack was contained within the HAZ for the entirety of the fracture test. Crack growth is from bottom to top in the image.

### ***Round Robin Testing at SNL-CA and SRNL***

A round robin study was performed at SNL-CA and SRNL to evaluate the test procedure developed at SNL. A set of four 21-6-9/308L HAZ bend samples were sent to SRNL for precracking in air. Samples were then evaluated by both SRNL and SNL-CA to ensure precracks were located in the HAZ, similar to the analysis performed and shown in Fig. 3b. Precracking procedures appear to be consistent between laboratories as the precrack lengths and locations in the HAZ were similar to previously tested 21-6-9/308L HAZ. Samples were hydrogen charged at SNL-CA and three were returned to SRNL for fracture testing, with one tested at SNL-CA. The fracture tests are expected to be completed in FY16 Q1.

### ***Established Test Plan for Tritium Exposed Specimens***

Stainless steel weld fusion zone and heat affected zone samples of 21-6-9 and 304L were supplied to SRNL for precracking, tritium exposure, and fracture testing. The test plan, as shown in Table 1, was designed to establish a testing effort with SRNL for multiple-year tritium exposures. The test matrix consists of triplicate tests to be performed on the weld fusion zone and heat affected zone for exposure times of 6 months, 12 months and 24 months. Specimens will be fracture tested following tritium exposure.

Table 1 –Test Plan for Tritium Exposed Stainless Steel Welds Bend Samples

Material	uncharged	H charged	Tritium exposure time			Total
			6 months	12 months	24 months	
Forged 304L/308L HAZ	3	3	3	3	3	15
Forged 304L/308L FZ	3	3	3	3	3	15
Forged 21-6-9/308L HAZ	3	3	3	3	3	15
Forged 21-6-9/308L FZ	3	3	3	3	3	15

### ***Summary and Future Work***

This project enabled SNL-CA to develop a procedure to evaluate the isotope-induced fracture of stainless steel weld fusion zones and heat affected zones. The procedure was observed to yield consistent results among replicate tests. A round robin set of tests was conducted at SNL-CA and SRNL to confirm that there is consistency between lab testing procedures. A test plan was developed which will allow multiple year tritium testing of stainless steel welds and HAZ at SRNL. Bend specimens were prepared at SNL-CA and supplied to SRNL for prercracking and tritium exposure. Tritium exposure of supplied weld specimens will begin in FY16. In addition, SRNL will develop Transmission Electron Microscopy (TEM) techniques for future characterization of material and helium bubble microstructures. Future testing at SNL-CA is focused on developing a means to evaluate the effects of temperature on hydrogen degraded welds and heat affected zones. Testing will be focused at -50 °C as low temperatures tend to enhance the negative effects of hydrogen.

[1] J.A. Ronevich, B.P. Somerday, C.W. San Marchi, D.K. Balch, “Fracture threshold measurements of hydrogen precharged stainless steel weld fusion zones and heat affected zones,” in Proceedings of the ASME 2015 Pressure Vessels and Piping Conference, July 19-23, 2015, Boston Massachusetts, USA. PVP2015-45432.

